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Docket 85501LMB  
Customer No. 01333

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**  
**BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES**

In re Application of

Krishnan Chari, et al

MAGENTA LOW  
FLUORESCENCE DYE FOR  
COATED OPTICAL BEAD  
RANDOM ARRAY DNA  
ANALYSIS

Serial No. 10/713,522

Filed November 14, 2003

Mail Stop APPEAL BRIEF-PATENTS  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA. 22313-1450

Group Art Unit: 1641

Examiner: Shafiqul Haq

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*Frieda Grinnell*  
Frieda Grinnell

*January 4, 2007*  
Date

Sir:

**APPEAL BRIEF TRANSMITTAL**

Enclosed herewith is Appellants' Appeal Brief for the above-identified application.

The Commissioner is hereby authorized to charge the Appeal Brief filing fee to Eastman Kodak Company Deposit Account 05-0225. A duplicate copy of this letter is enclosed.

Respectfully submitted,

*Lynne M. Blank*  
\_\_\_\_\_  
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Enclosures

If the Examiner is unable to reach the Applicant(s) Attorney at the telephone number provided, the Examiner is requested to communicate with Eastman Kodak Company Patent Operations at (585) 477-4656.



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*Frederick D. Gannell*

*January 4, 2007*  
Date

Sir:

**APPEAL BRIEF PURSUANT TO 37 C.F.R. 41.37 and 35 U.S.C. 134**



## Table of Contents

<u>Table of Contents</u> .....	i
<u>Real Party In Interest</u> .....	1
<u>Related Appeals And Interferences</u> .....	1
<u>Status Of The Claims</u> .....	1
<u>Status Of Amendments</u> .....	1
<u>Summary of Claimed Subject Matter</u> .....	2
<u>Grounds of Rejection to be Reviewed on Appeal</u> .....	2
<u>Arguments</u> .....	2
<u>Summary</u> .....	12
<u>Conclusion</u> .....	13
<u>Appendix I – Claims On Appeal</u> .....	14
<u>Appendix II – Evidence</u> .....	17
<u>Appendix III – Related Proceedings</u> .....	18



## **APPELLANT'S BRIEF ON APPEAL**

Appellants hereby appeal to the Board of Patent Appeals and Interferences from the Examiner's Final Rejection of claims 1-11 which was contained in the Office Action mailed 06/02/2006.

A timely Notice of Appeal was filed 08/29/2006.

### **Real Party In Interest**

As indicated above in the caption of the Brief, the Eastman Kodak Company is the real party in interest.

### **Related Appeals And Interferences**

No appeals or interferences are known which will directly affect or be directly affected by or have bearing on the Board's decision in the pending appeal.

### **Status Of The Claims**

Claims 1-33 are pending in the application.

Claims 12-33 stand withdrawn from consideration as directed to a non-elected invention, pursuant to a restriction requirement made by the Examiner in an Office Action dated 11/17/2005, and the Applicant's election made in the Response dated 3/17/2006.

Claims 1-11 are being appealed.

Appendix I provides a clean, double spaced copy of the claims on appeal.

### **Status Of Amendments**

An Amendment After Final was filed on August 1, 2006, subsequent to the Final Rejection. An Advisory Action dated August 22, 2006 was then received not entering the proposed amendment.

### **Summary of Claimed Subject Matter**

The invention relates to a random array of colored microspheres (page 13, lines 9-11) and a dye for coloring microspheres magenta (page 6, lines 6-7). The dyes are suitable for coloring polystyrene beads (page 7, lines 9-11) and have

good solubility in solvents (page 7, lines 9-11). The colorant materials have very low fluorescence intensity (page 6, lines 6-7) such that the resultant colored microspheres do not substantially fluoresce when excited by visible light (page 7, lines 11-14).

Independent claim 1 recites a coating composition for making a protein microarray (page 13, lines 17-19), the composition is made up of a gelling agent or a precursor to a gelling agent (page 13, lines 14-16), and microspheres (page 13, lines 9-14); the microspheres containing a low fluorescing dye (page 6, lines 1-3) represented by Formula (I) (page 6, lines 10-16).

Dependant claim 2 recites a coating composition for making a protein microarray (page 13, lines 17-19), the composition is made up of a gelling agent or a precursor to a gelling agent (page 13, lines 14-16), and microspheres (page 13, lines 9-14); the microspheres containing a low fluorescing dye (page 6, lines 1-3) represented by Formula (II) (page 7, lines 1-5).

### **Grounds of Rejection to be Reviewed on Appeal**

The following issues are presented for review by the Board of Patent Appeals and Interferences:

1. Whether claims 1-3 and 9-10 are anticipated under 35 U.S.C. § 102(b) as being anticipated by Chen et al. (EP 1127707 A1).
2. Whether claims 1-3 are anticipated under 35 U.S.C. § 102(b) as being anticipated by Evans et al. (U.S. 4,420,550).
3. Whether claims 1-11 are unpatentable under 35 U.S.C. § 103(a) over Noonan et al. (U.S. 5,334,575) in view of Evans et al. (U.S. 4,420,550).
4. Whether claims 1-11 are unpatentable under 35 U.S.C. § 103(a) over Qiao et al. (U.S. 6,916,620) in view of Evans et al. (U.S. 4,420,550).
5. Whether claims 1-11 are unpatentable under 35 U.S.C. § 103(a) over Qiao et al. (U.S. 7,011,971) in view of Evans et al. (U.S. 4,420,550).

### **Arguments**

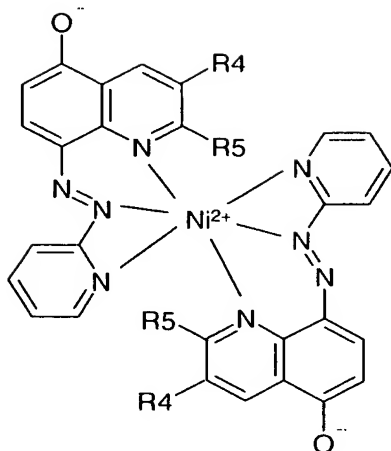
#### **Rejection of claims 1-3 and 9-10 under 35 U.S.C. § 102(b) over Chen et al.**

In section 9 of the Final Office action mailed June 2, 2006 claims 1-3 and 9-10 stand rejected under 35 U.S.C. § 102(b) as being anticipated over Chen et al. (EP 1127707 A1). The Examiner asserts that Chen et al. discloses a coating composition comprising a dye-containing polymeric latex and gelling agent and at least one of the nickel metallized dye of the reference anticipates the dyes of present application, for claims 9-10, Chen et al. disclose the microspheres to be comprised of synthetic polymeric materials. With respect to recitation "low fluorescent dye represented by the formula (I)", the low fluorescence property is inherent in the dye of Chen et al. because water-insoluble Dye-1 of Chen et al. reads on Formula (II) of instant application and both the dyes have the same structure and are expected to have the same property.

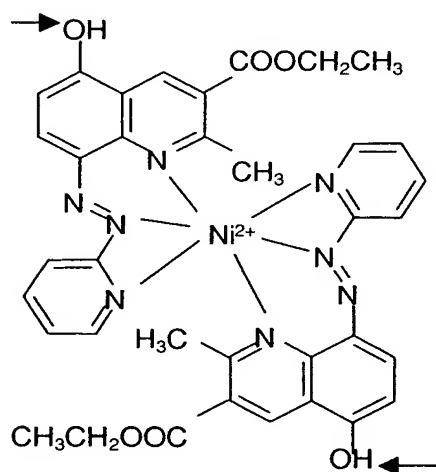
This rejection is respectfully urged as in error and reversal is requested for the following reasons.

Chen et al. discloses a coating composition comprising a dye-containing polymeric latex, gelling agent nickel metallized dye. However, Chen et al. fails to disclose a low fluorescing dye of Formula (I) or (II) as claimed by the instant invention. Additionally, low fluorescence is not an inherent property of the dyes disclosed by the Chen et al. reference. Chen et al. discloses that a broad range of water-insoluble dyes may be used such as oil, disperse, solvent, or metal-complex dyes. (Para. [0010]). The instant invention illustrates that Comparative dyes 1-4 (Page 21, Table 1) have high fluorescence values, and are outside the scope of the claimed invention. Specifically, Comparative dye 3 (Page 21, Table 1) is a disperse azo dye that is disclosed by Chen et al. as an acceptable water-insoluble dye to be utilized by the reference (Para. [0010]). By contrast, disperse azo dyes are inapplicable for use in the instant invention as they demonstrate a high fluorescence value as demonstrated by Comparative dye 3. Chen et al. fails to disclose any distinction between low fluorescing metal-complex dyes as claimed by the instant invention and high fluorescing disperse dyes, as in Comparative dye 3.

Additionally, the inherency doctrine is misapplied in the current rejection. Formula (II) of instant application appears as follows:



while the dye of Chen et al. has the following structure:



As can be seen, the two structures are different. Chen et al. fails to set forth every claimed limitation of the instant invention. The reference fails to disclose Formula (I) or (II) of the instant application. To modify the dye of Chen et al. to arrive at Formula (I) or (II) of the instant invention requires an obviousness type argument under U.S.C. § 103. Inherency is proper when interpreting the characteristics of similar chemical structures, not for modifying a chemical

structure into a different chemical structure to arrive at a claimed limitation.

Thus, the basis for applying the inherency doctrine is not present.

Furthermore, even applying the inherency doctrine the reference fails to anticipate the instant invention. The fact that a certain characteristic may be present in the prior art is not sufficient to establish the inherency of that characteristic. *In re Rijckaert*, 9 F.3d 1531, 1534 (Fed. Cir. 1993). "Inherency, however, may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient." *In re Robertson*, 169 F.3d 743, 745 (Fed. Cir. 1999). As a result, the reference fails to anticipate the present claims.

**Rejection of claims 1-3 under 35 U.S.C. § 102(b) over Evans et al.**

In section 10 of the Final Office action mailed June 2, 2006 claims 1-3 stand rejected under 35 U.S.C. § 102(b) as being anticipated over Evans et al. (U.S. 4,420,550). The Examiner asserts that Evans et al. discloses a coating composition comprising a dye-containing emulsion and gelling agent, and at least one of the nickel metallized dye of the reference anticipates the dyes of present application. The Examiner indicates that "microspheres" by definition are suspensions when dispersed in liquid and therefore, "emulsion" which is a "suspension" of small globules on one liquid with which the first will not mix, fits the definition of "microspheres."

This rejection is respectfully urged as in error and reversal is requested for the following reasons.

Evans et al. discloses certain non-diffusible magenta dye-releasing compounds which, as a function of development of a silver halide emulsion layer, release a diffusible magenta dye. However, the reference fails to disclose microspheres, let alone a dye contained in a microsphere as claimed by the instant invention. In addition, the reference is silent with respect to low fluorescing dyes.

A claim is anticipated only if each and every element as set forth in the claim is found either expressly or inherently described in a single prior art reference. The identical invention must be shown in as complete detail as is contained in the claim.



The term "microsphere" is clearly defined as "a minute sphere" by the Merriam-Webster Online Dictionary. The term "sphere" is defined as a globular body, such as a ball or a solid that is bounded by a surface consisting of all points at a given distance from a point constituting its center by the Merriam-Webster Online Dictionary. Given the plain meaning of the term "microsphere" it is clear that the term refers to a particle that is spherical. Words of a claim must be given their "plain meaning" unless they are defined otherwise in the specification. MPEP § 2111.01. Ordinary, simple English words, whose meaning is clear and unquestionable, absent any indication that their use in a particular context changes their meaning, are construed to mean exactly what they say. *Chef America, Inc. v. Lamb-Weston, Inc.*, 358 F.3d 1371, 1372, 69 USPQ2d 1857 (Fed. Cir. 2004). Silver halide "emulsions" are not microspheres as they are not spherical in shape. A silver halide "emulsion" is actually not an emulsion at all, but instead it is a dispersion. "As already noted, the silver halides used in photography are dispersions of microscopic crystals in a colloidal binder that is usually bone gelatin. Although such dispersions are referred to as emulsions or photographic emulsions, they are really dispersions." See Attachment 1, previously submitted with the response dated March 17, 2006 (page 3, lines 1-4). Also, referring to Attachment 1 previously submitted, page 5, silver halide grains are not spherical, and therefore, are not solids bounded by a surface consisting of all points at a given distance from a point constituting its center. As the silver halide "emulsions" disclosed in Evens et al. are not spherical the reference fails to disclose microspheres and therefore, fails to disclose all of the claimed limitations of the instant invention.

Additionally, Evens et al. is silent with respect to low-fluorescing dyes as claimed in the instant invention. Therefore, it is respectfully urged that the reference fails to disclose all of the claimed limitations of the instant invention.

**Rejection of claims 1-11 under 35 U.S.C. § 103(a) over Noonan et al. in view of Evans et al.**

In section 12 of the Final Office action mailed June 2, 2006 claims 1-11 stand rejected under 35 U.S.C. § 103(a) as being unpatentable over Noonan et al. (U.S. 5,334,575) in view of Evans et al. (U.S. 4,420,550). The Examiner asserts

that Noonan et al. discloses a coating composition comprising a gelling agent and microspheres containing a magenta dye. The Examiner further indicates that Noonan et al. fails to disclose nickel metallized dye of Formula (I) or (II) as claimed in the instant invention. The Examiner relies on Evens et al. to teach this claimed feature.

This rejection is respectfully urged as in error and reversal is requested for the following reasons.

Noonan et al. relates to the use of certain dye-containing beads in the donor element of a laser-induced thermal dye transfer system, specifically a monochrome dye donor element for laser-induced thermal dye transfer. Noonan et al. teaches a support having a dye layer comprising solid, homogeneous beads which contain an image dye, a binder and a laser light-absorbing material.

Evans relates to photography and more particularly to color diffusion transfer photography employing certain non-diffusible magenta dye-releasing compounds which, as a function of development of a silver halide emulsion layer, release a diffusible magenta dye. The dye-releasing compound can be premetallized or a metal complex of the released dye can be formed in an image-receiving layer.

The present invention relates to a coating composition for making a protein microarray, the composition comprising a gelling agent or a precursor to a gelling agent, and microspheres; the microspheres containing a low fluorescing dye represented by the Formula (I).

To establish a prima facie case of obviousness, there must be some suggestion or motivation in the reference or in the general knowledge available to one skilled in the art to modify the reference, there must be a reasonable expectation of success, and the prior art reference must teach or suggest all the claim limitations.

As noted by the Examiner Noonan et al. fails to teach a nickel metallized dye of Formula (I) or (II) as claimed by the instant invention. As discussed above, Evans discloses a silver halide "emulsion" which is truly a dispersion, not an emulsion, and, hence, not a microsphere, and further fails to mention a dye contained in a microsphere. Neither reference, alone or in combination, teaches

low fluorescing dyes of Formula I or II as claimed by the present invention. Noonan et al. discloses light absorbing material however, fails to teach or suggest a low fluorescing dye as claimed in the instant invention. A material may have the possess both high light absorbing and high florescence characteristics. A material can absorb light and later emit light at a different wavelength increasing the fluorescence of the material while maintaining a high absorbance, such as those disclosed in WO 02/077291.

The present invention provides a microarray that is less costly and easier to prepare than those previously disclosed, and further can be used in a colored microarray device wherein green light absorbance is desired to be maximized and fluorescence of the dye imbibed in the colored polystyrene microsphere bead is desired to be minimized. The references are silent with respect to the control of fluorescence levels in a microsphere and therefore, provide no likelihood of success in the use or identification of a low fluorescing dye of Formula (I) or (II). There are a very large number of compounds known to those skilled in the art, which may be utilized as dyes. There are, further, a tremendous number of types of microspheres. Microarray systems are very complex and unpredictable and the fact that two technologies are independently successful does not indicate that the combination will be useful or beneficial. As indicated in the present specification,

there are no general guideline parameters with which a colorant scientist may predict the fluorescence of any given colorant material. Therefore, the colorant scientist must undertake an empirical approach to the discovery of colorant materials that are non-fluorescent. It appears that dye materials containing a specific halogen functionality are particularly likely to possess the property of very low fluorescence. Thus, the dyes of this invention have been found to have good solubility in the organic solvents required for bead coloration, high extinction, and remarkably low fluorescence when imbibed in a polystyrene microsphere bead. (pg. 5, line 28 - pg. 6, line 5).

At most, the Examiner has set forth an argument that it would be "obvious to try" the combination of the cited references. Therefore, there is no reasonable expectation of success found in any combination of the cited references.

The present invention provides surprising results, as dye materials containing a specific halogen functionality are particularly likely to possess the

property of very low fluorescence. As indicated on pg. 21, Table I of the specification, the presently claimed dyes of Formula I are low fluorescing, when compared to other, similar dyes.

**Rejection of claims 1-11 under 35 U.S.C. § 103(a) over Qiao et al. ('620) in view of Evans et al.**

In section 13 of the Final Office action mailed June 2, 2006 claims 1-11 stand rejected under 35 U.S.C. § 103(a) as being unpatentable over Qiao et al. (U.S. 6,916,620) in view of Evans et al. (U.S. 4,420,550). The Examiner asserts that Qiao et al. ('620) discloses a microarray coating composition comprising a gelling agent and microsphere (beads) containing a magenta dye, as for claims 6-8, Qiao et al. ('620) discloses beads size of 1 to 50 microns and as for claims 9-11, Qiao et al. ('620) discloses the beads comprising polystyrene. The Examiner indicates that Qiao et al. ('620) discloses magenta dye but fail to disclose nickel metallized dye of Formula (I) and (II) of present application, however, Evans et al. discloses a coating composition comprising magenta dye of Formula (I) and (II) of present application. Evans et al. also discloses that the metallized dye have less unwanted absorption than other metallizable dyes, and, therefore, given the above fact that magenta dye of present application is known in the art as a coating composition and is useful for its less absorptive properties, it would have been obvious at the time of the invention to a person of ordinary skill in the art to substitute equivalent magenta dye of Evans et al. in the coating composition of Qiao et al. ('620) with the expectation of obtaining a similarly useful microarray coating composition.

This rejection is respectfully urged as in error and reversal is requested for the following reasons.

The references fail to teach or suggest the modification to produce the presently claimed invention. Qiao et al. ('620) discloses an assay method relying on the detection of fluorescence or chemiluminescence (Abstract) and specifically exemplifies Dye 1 in col. 10, lines 5-20, utilized in inventive Formulation 1 (col. 9, lines 6-34). Dye 1 is equivalent to Comparative dye 4 of the present invention, which as disclosed on pg. 23, Table 1 has a high fluorescence emission. As discussed above, Evans et al. discloses a silver halide "emulsion" which is truly a dispersion,

not an emulsion, and, hence, not a microsphere. Neither reference, alone or in combination teaches or suggests low fluorescing dyes of Formula (I) or (II) as claimed in the instant invention.

The references are silent with respect to the control of fluorescence levels in a microsphere and therefore, provide no likelihood of success in the use or identification of a low fluorescing dye of Formula (I) or (II). As discussed above, there are a very large number of compounds known to those skilled in the art, which may be utilized as dyes and at most, the Examiner has set forth an argument that it would be "obvious to try" the combination of the cited references. Therefore, there is no reasonable expectation of success found in any combination of the cited references.

Furthermore, as discussed above the present invention provides surprising results, as the dye materials of the instant invention possess the property of very low fluorescence when compared to other, similar dyes.

**Rejection of claims 1-11 under 35 U.S.C. § 103(a) over Qiao et al. ('971) in view of Evans et al.**

In section 14 of the Final Office action mailed June 2, 2006 claims 1-11 stand rejected under 35 U.S.C. § 103(a) as being unpatentable over Qiao et al. (U.S. 7,011,971) in view of Evans et al. (U.S. 4,420,550). The Examiner asserts that Qiao et al. ('971) discloses a microarray coating composition comprising a gelling agent and microsphere (beads) containing a magenta dye, as for claims 6-8, Qiao et al. ('971) discloses beads size of 1 to 50 microns and as for claims 9-11, Qiao et al. ('971) discloses the beads comprising polystyrene. The Examiner indicates that Qiao et al. ('971) discloses magenta dye but fail to disclose nickel metallized dye of Formula (I) and (II) of present application, however, Evans et al. discloses a coating composition comprising magenta dye of Formula (I) and (II) of present application. Evans et al. also discloses that the metallized dye has less unwanted absorption than other metallizable dyes, and, therefore, given the above fact that magenta dye of present application is known in the art as coating composition and is useful for its less absorptive properties, it would have been obvious at the time of the invention to a person of ordinary skill in the art to substitute an equivalent magenta dye of Evans et al. in the coating composition of

Qiao et al. ('971) with the expectation of obtaining a similarly useful microarray coating composition.

This rejection is respectfully urged as in error and reversal is requested for the following reasons.

As discussed above, Dye 1 on col. 7, lines 27-43 is utilized in Formulation 1 of Qiao et al. ('971) (col. 6, lines 19-53). Dye 1 is equivalent to Comparative dye 4 of the present invention, which as disclosed on pg. 23, Table 1 has a high fluoresce emission. As discussed above, Evans et al. discloses a silver halide "emulsion" which is truly a dispersion, not an emulsion, and, hence, not a microsphere. Neither reference, alone or in combination teaches or suggests low fluorescing dyes of Formula (I) or (II) as claimed in the instant invention.

Also as discussed above, there is no reasonable expectation of success found in any combination of the cited references. The references are silent with respect to the control of fluorescence levels in a microsphere. There are a very large number of compounds known to those skilled in the art, which may be utilized as dyes and at most, the Examiner has set forth an argument that it would be "obvious to try" the combination of the cited references.

Furthermore, as discussed above the present invention provides surprising results, as the dye materials of the instant invention possess the property of very low fluorescence when compared to other, similar dyes.

**Examiner's response to Applicant's arguments**

In the Advisory Action mailed August 22, 2006 the Examiner indicates that the rejections under 35 U.S.C. § 112 have been overcome.

The Examiner reasserts that Chen et al. reads on Formula (II) of the instant invention. However, as discussed above, the chemical formula disclosed in Chen et al. is distinct from that claimed by the instant invention.

The Examiner further asserts that Evens et al. discloses halide emulsions which fall under the definition of microsphere. However, as discussed above and referring to Attachment 1, silver halide emulsions are actually dispersions and therefore, are not microspheres as claimed by the instant invention.

The Examiner indicates that it would have been obvious to substitute magenta dyes as disclosed in Qiao et al. with the nickel metallized magenta dye of

Evans et al. to decrease unwanted absorption with a reasonable expectation of success. However, as discussed above, the references are silent with respect to the control of fluorescence levels in a microsphere and instead focus on controlling absorption, an entirely distinct property.

### **Summary**

Chen et al. fails to disclose a low fluorescing dye of Formula (I) or (II) as claimed by the instant invention. Chen et al. discloses a broad range of water-insoluble dyes and fails to distinguish between low fluorescing metal-complex dyes as claimed by the instant invention and high fluorescing disperse dyes as claimed by Chen et al. Furthermore, Chen et al. does not disclose Formula (I) or (II) as claimed by the instant invention, and therefore fails to anticipate the instant invention.

Evens et al. fails to disclose microspheres as claimed by the instant invention. As noted in Attachment 1 silver halide "emulsions" are actually dispersions and not spherical in nature. Additionally, Evens et al. is silent regarding low-fluorescing dyes as claimed by the instant invention. For these reasons the reference fails to anticipate the present invention as claimed.

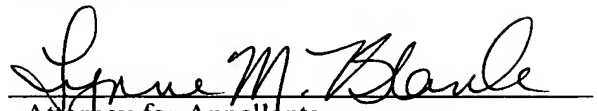
As noted by the Examiner Noonan et al. fails to teach a nickel metallized dye of Formula (I) or (II) as claimed by the instant invention. Furthermore, Noonan et al. is silent regarding low-fluorescing dyes as claimed by the present invention. Noonan et al. discloses a high absorbing material however, the instant invention claims a low-fluorescing dye. A material may absorb most of the light at one wavelength and still fluoresce a large amount of light at a different wavelength, resulting in a material with high absorption but also high fluorescence. Qiao et al. discloses an inventive dye which is equivalent to comparative dye 1 of the instant invention, which demonstrates a high fluorescence. There is no motivation to combine any of the above references as none of the references disclose a low fluorescing material. At most, the rejection is set forth that it would be "obvious to try" the combination of the cited references. The references lack any motivation to suggest combination to arrive at

a low fluorescing dye of Formula (I) or (II) as claimed by the instant invention.  
Therefore, reversal is respectfully requested.

**Conclusion**

For the above reasons, Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the rejection by the Examiner and mandate the allowance of Claims 1-11.

Respectfully submitted,

A handwritten signature in cursive script, appearing to read "Lynne M. Blank", is written over a horizontal line.

Attorney for Appellants  
Registration No. 42,334

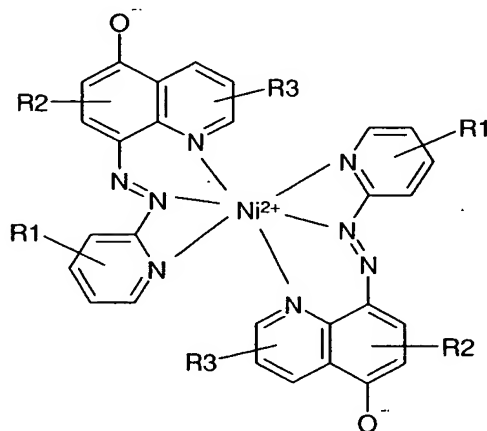
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Enclosures

If the Examiner is unable to reach the Applicant(s) Attorney at the telephone number provided, the Examiner is requested to communicate with Eastman Kodak Company Patent Operations at (585) 477-4656.



## Appendix I - Claims on Appeal

1. A coating composition for making a protein microarray, the composition comprising a gelling agent or a precursor to a gelling agent, and microspheres; the microspheres containing a low fluorescing dye represented by the Formula (I):



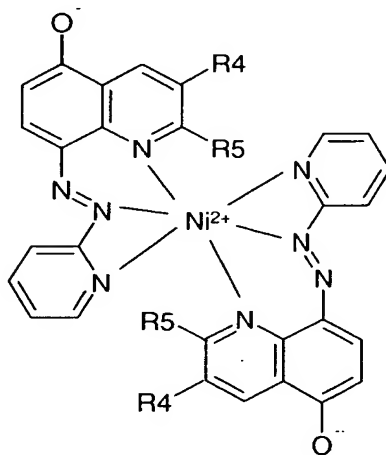
Formula (I)

wherein:  $\text{R}_1$  = one or more substituent selected from the group of H, chloro, alkoxy carbonyl, arylsulfamoyl, or alkylsulfamoyl;

$\text{R}_2$  = one or more substituent selected from the group of H, carboxamido, or alkoxy carbonyl;

$\text{R}_3$  = one or more substituent selected from the group of H, chloro, substituted or unsubstituted alkyl, aryl, carboxamido, or alkoxy carbonyl.

2. The coating composition according to claim 1 wherein the microspheres contain a dye represented by the Formula (II)



Formula(II)

wherein:  $\text{R}_4$  = alkoxycarbonyl;  
 $\text{R}_5$  = H, chloro, or alkyl.

3. The coating composition according to claim 1 wherein the gelling agent is gelatin.

4. The coating composition according to claim 1 wherein the gelling agent undergoes thermal gelation.

5. The coating composition according to claim 3 wherein the gelatin is alkali pretreated gelatin.

6. The coating composition according to claim 1 wherein the microspheres have a mean diameter between 1 and 50 microns.

7. The coating composition according to claim 1 wherein the microspheres have a mean diameter between 3 and 30 microns.

8. The coating composition according to claim 1 wherein the microspheres have a mean diameter between 5 and 20 microns.

9. The coating composition according to claim 1 wherein the microspheres comprise a synthetic or natural polymeric material.

10. The coating composition according to claim 9 wherein the polymeric material is an amorphous polymer.

11. The coating composition according to claim 10 wherein the amorphous polymer is polystyrene.

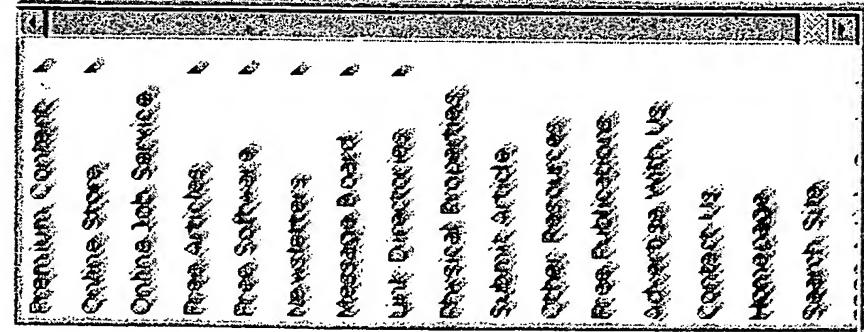
## **Appendix II - Evidence**

Attachment 1 has been previously submitted on the response dated March 17, 2006. The Evidence includes:

Attachment 1: An article on the chemistry of photography by dr. Drew Myers found at <http://www.cheresources.com/photochem.shtml>.

### **Appendix III – Related Proceedings**

None



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## Chemistry of Photography

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While it is easy to make comparisons between the pupil of the eye and the f-stop of a camera or between the retina of the eye and photographic film, once we get past the basic similarities of the optics of the two systems, comparisons begin to rapidly break down. The eye is not only much more complex than a camera and its film, but the two imaging devices function by different chemical mechanisms. The photographer (or the automatic exposure system of the camera) regulates the f-stop opening and time of exposure of her camera to match the sensitivity of film, while the iris and retina sensitivity of the eye adjust to correspond to the light level of the scene.

While science is slowly putting together the pieces to explain the functioning of our vision system, the basic nuts and bolts of classical photography have been known for years, although certain details remain the subject of some discussion. Just as in the human eye, classical photographic systems are composed of two separate, but interrelated processes – the basic black and white image structure and the finer points of color reproduction.

This first installment on the chemistry of photography is intended to introduce, in a simplified way, the basic concepts of silver halide photography. It will not delve into the physics of optics, the functioning of cameras and lenses, photographic techniques, non-silver processes, or the artistic aspects of photography. Nor will it go beyond a cursory mention of color photographic processes, which will be left for the future. Anyone interested in more detail is referred to the selected bibliographic material cited at the end.

## A Brief History of Black and White Silver Halide Photography

Attachment 1  
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Perhaps the earliest reference to the concept of silver-based black and white photography is that of J. H. Schulze who observed in 1727 that a mixture of silver nitrate and chalk darkened on exposure to light. The first semi-permanent images were obtained in 1824 by Nicéphore Niepce, a French physicist, using glass plates coated with a dispersion of silver salts in bitumen (a coal derivative). In the early 1830's, Niepce's partner, Louis Daguerre, discovered by accident that mercury vapor was capable of developing an image on a silver-plated copper sheet that had been previously sensitized by iodine vapor. The image, which was called a daguerreotype, could be made permanent by washing the plate with hot concentrated salt solution. In 1889 Daguerre demonstrated his photographic process to the Academy of Sciences in Paris. The process was later improved by using sodium thiosulfate to wash off the unexposed silver salts.

In 1841, an Englishman, William Henry Fox Talbot introduced a new system, the calotype process. The Talbot process involved a paper than had been sensitized to light by a coating of silver iodide. A negative image was produced on the exposed light-sensitive paper by bathing it in a solution of gallic acid in a development process essentially the same as that used today. If the paper base employed was semitransparent, the original negative image could be laid over another piece of sensitized paper which, when exposed and developed, yielded a "positive," or direct copy of the original. The process would be equivalent to what is termed "contact printing" today. Although the calotype process required less time than that of Daguerre, the Talbot images were not particularly sharp because of the fluidity of the medium employed to suspend the silver iodide crystals.

Originally, the silver salts were held on glass using egg white as a binder. This provided relatively sharp images although they were easily damaged. By 1871, the problem had been solved by Dr. R. L. Maddox, an amateur photographer and physician, who discovered a way to prepare gelatin dispersions of silver salts on glass plates. In 1887 George Eastman introduced the Kodak system in which a silver halide-in-gelatin dispersion was coated on a cellulose nitrate base and loaded into a camera. The camera could take 100 pictures and when all were exposed, camera and film were returned to Rochester, New York, for processing. With those innovations the age of modern photography had arrived.

## Photochemistry of Silver Salts

To understand the fundamental chemistry of silver-based photography, we must look at the photochemistry of silver salts. A typical photographic film contains tiny crystals of very slightly

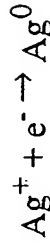
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soluble silver halide salts such as silver bromide (AgBr) commonly referred to as "grains." The grains are suspended in a gelatin matrix and the resulting gelatin dispersion, incorrectly (from a physical chemistry standpoint), but traditionally referred to as an "emulsion," is melted and applied as a thin coating on a polymer base or, as in older applications, on a glass plate.

Figure 1 shows a schematic representation of the silver halide process. When light or radiation of appropriate wavelength strikes one of the silver halide crystals, a series of reactions begins that produces a small amount of free silver in the grain. Initially, a free bromine atom is produced when the bromide ion absorbs the photon of light:



The silver ion can then combine with the electron to produce a silver atom.



Association within the grains produces species such as  $\text{Ag}_2^+$ ,  $\text{Ag}_2^0$ ,  $\text{Ag}_3^+$ ,  $\text{Ag}_3^0$ ,  $\text{Ag}_4^+$ , and  $\text{Ag}_4^0$ . The free silver produced in the exposed silver halide grains constitutes what is referred to as the "latent image," which is later amplified by the development process.

The grains containing the free silver in the form of  $\text{Ag}_4^0$  are readily reduced by chemicals referred to as "developers" forming relatively large amounts of free silver; that deposit of free silver produces a dark area in that section of the film. The developer under the same conditions does not significantly affect the unexposed grains.

The radiation or light sensitivity of a silver halide film (referred to in the trade as its "speed" and denoted on commercial film as its ASA in the United States or DIN in Europe) is related to the size of the grain and to the specific halide composition employed. In general, as the grain size in the emulsion increases, the effective light sensitivity of the film increases - up to a point. An optimum value of grain size for a given sensitivity is found to exist because the same number of silver atoms are needed to initiate reduction of the entire grain by the developer despite the grain size, so that producing larger grains reaches a point of diminishing returns and no further benefit is obtained.



All photographic emulsions contain crystals of varying sizes, but within a given emulsion the range is from less than 0.1 micron in slow emulsions (e.g., for paper prints) to a few microns in "fast" negative emulsions.

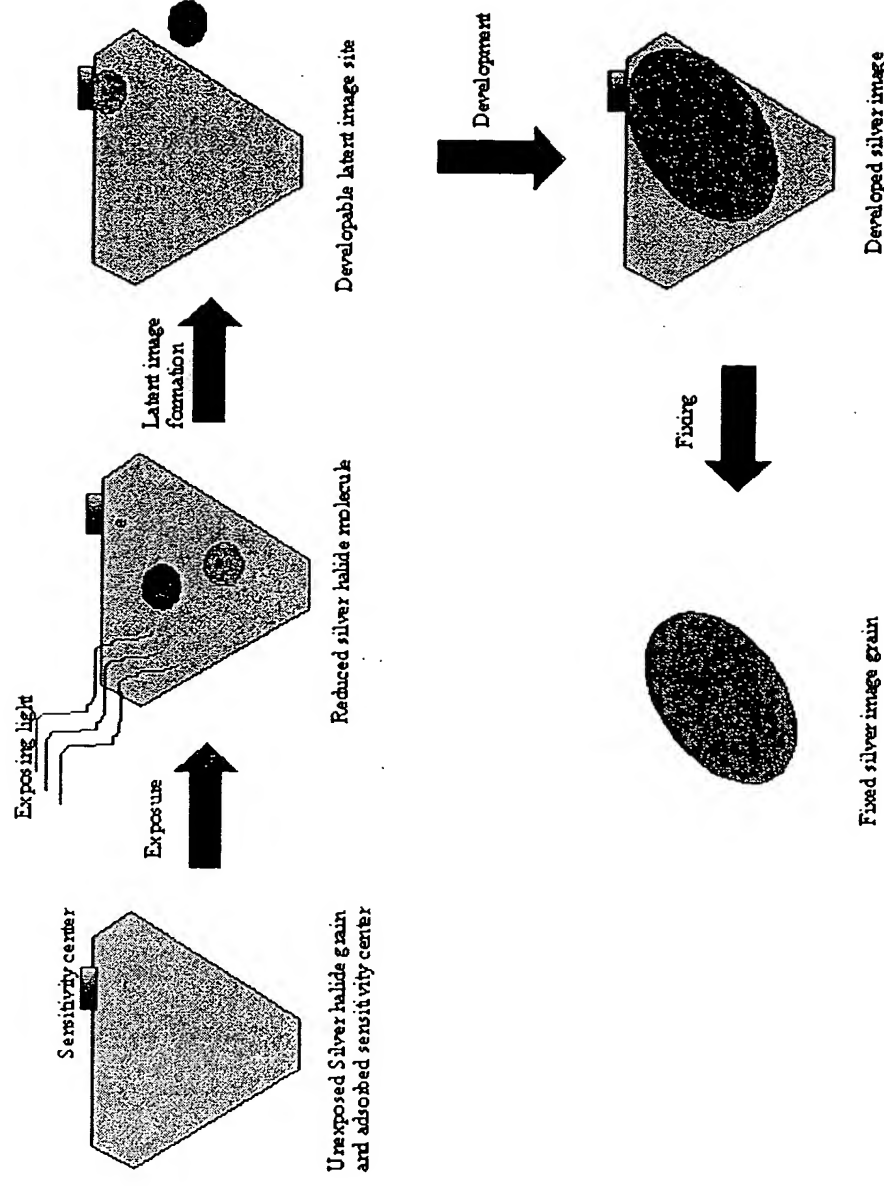


Figure 1: A Simplified Schematic Representation of the Silver Halide Process

An interesting modern innovation in photographic emulsion technology is related to the basic concept of silver halide grain geometry. In a classical silver halide crystal, typically a cubic crystal lattice, the structure will be relatively symmetrical in that the orientation of the crystal in the coated film will always present the same approximate surface area to be exposed. Extensive research efforts led to the development of grain precipitation processes that produced flatter "tablet" grains in which the crystals possessed a more asymmetric geometry, and in which a

larger surface area was presented for exposure for the same given weight of silver halide (Fig. 2). That development resulted in significant improvements in film sensitivity and reductions in the amount of silver needed to obtain a given sensitivity – and a potentially important reduction in the cost of the film.

### The Latent Image and Image Development

The silver halide process is by far the most important of all of the radiation-sensitive photographic systems in use today. The principal reason for this superiority is the high sensitivity of the system – the amount of radiant energy required to produce a useful image – and the extreme flexibility of the system in terms of adjusting sensitivity, contrast, tonal range and other such aspects of the product. The impact of a single photon on a silver halide grain, for example, produces a nucleus of at least four silver atoms, and that effect can be amplified as much as a billion times by the action of a properly chosen reducing agent or "developer."

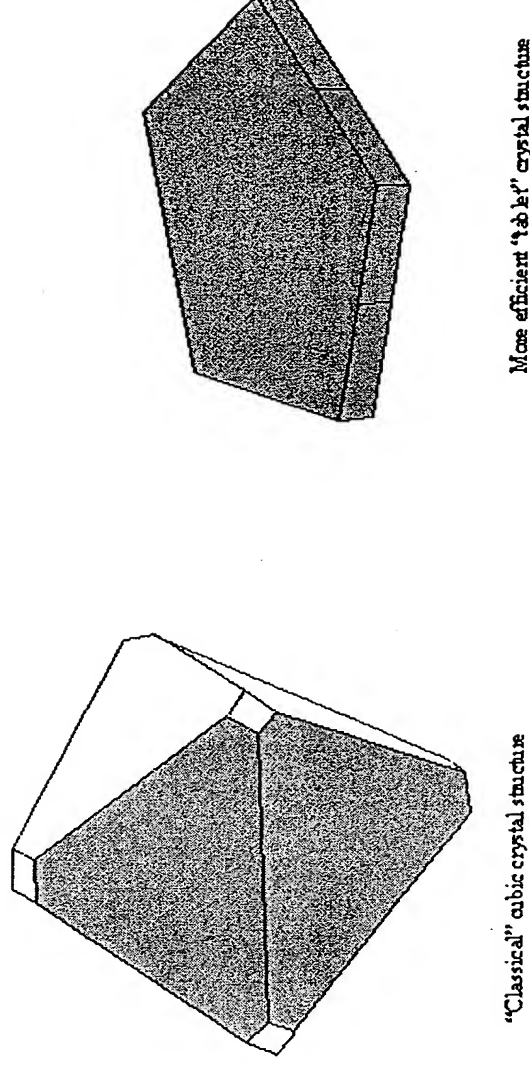


Figure 2: A Schematic Representation of Cubic and "Tablet" Silver Halide Grains

The silver halides employed are silver bromide, silver chloride and silver iodide. The first two may be used separately or combined, depending on the sensitivity and tonal qualities desired in

the product. Silver iodide is always combined with silver bromide or silver chloride.

As already noted, the silver halides used in photography are dispersions of microscopic crystals in a colloidal binder that is usually bone gelatin. Although such dispersions are referred to as emulsions or photographic emulsions, they are really dispersions.

When an exposed film is placed in a developer solution, the grains that contain silver nuclei are reduced much faster than those that do not. The more nuclei present in a given grain (i.e., the greater the exposure of that grain), the faster the reaction with developer and the darker the image at that site in the film. Factors such as temperature, concentration of the developer, pH, and the total number of nuclei in each grain determine the extent of development and the intensity of free silver (blackness) deposited in the film emulsion in a given time.


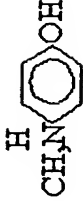


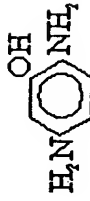
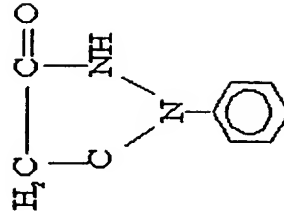
Not only must the developer be capable of reducing silver ions to free silver, but it must be selective enough not to reduce the unexposed grains, a process known as "fogging," within the time frame of the development process. Some substances that are commonly used as silver halide developers are listed in Table 1.

The developer is oxidized in the process. If not properly protected it can also be oxidized by air, a process that, if not prevented, will result in the loss of developer activity. To help prevent such effects, commercial developer solutions commonly contain preservatives such as sodium sulfite.

### **Gelatin – The Film Matrix**

Gelatin is a protein extracted from animal hides, bone, and sinew that belongs to the class of substances known as hydrophilic ("water loving") polymers, which also includes other proteins, gums, starches, and a wide variety of synthetic polymers. Photographic grade gelatin is usually produced by an alkaline extraction process using bovine bones, although some acidic processes have been developed. Some special gelatins are also used that are derived from pigskins. The origin and quality of the raw materials used in the gelatin process, the conditions employed (pH, temperature, time, etc.), and the presence or absence of certain possible contaminants (e.g., mercury or other heavy metals, lipids, sulfur compounds, etc.) are of vital importance to the production of material suitable for use in modern photographic systems. In fact, photographic gelatin is much purer than that used in food applications.

Table 1: Common Silver Halide Developers

Name	Structure
p-Dihydroxybenzene (Hydroquinone)	
Monomethylp-aminophenol (Metol or Elon)	
1,2,3-Trihydroxybenzene (Pyrogallol)	
p-Aminophenol	
Diaminophenol (Amidol)	
1-Phenyl-3-pyrazolidone	

Gelatin solutions have the useful property of behaving as a liquid when warm and setting to a relatively hard gel when cool. When coated on a substrate and dried it is flexible and reasonably resistant to physical damage, but it readily absorbs water (as in the development process). Generally, the coated gelatin dispersion will contain some material such as formaldehyde as a "hardener" that serves to produce a limited number of cross links among protein chains that

improve the physical characteristics of the coated material. The gelatin swells rapidly, absorbing water and dissolved development chemicals, but it does not dissolve or disintegrate at normal temperatures. These properties are essential in the preparation of a photographic emulsion, in coating it on film base or paper, and in the processes of development, fixation and washing.

Gelatin is more than a means of holding the sensitive silver halide salts in place on a film base, however. It is an integral part of the process and can significantly affect the properties of the final photographic product. Controlling the characteristics of the gelatin is absolutely essential to the photographic emulsion preparation, the process of coating the emulsion on the film base, the adhesion of the coated material to the film base, the wetting characteristics of the coated material in the development process, the physical characteristics of the dried developed product, and the long-term stability of the developed image. In a way, one could safely say that the gelatin is almost as important as silver to the overall photographic system.

In the preparation of a photographic emulsion the gelatin also acts as an anti-coagulant or stabilizing colloid. The silver halide formed in fluid gelatin does not precipitate out of solution but remains uniformly distributed throughout the preparation, ripening (see below), and coating processes. The gelatin is an important factor in determining the dispersity or range of grain sizes of the silver halide. By suitable regulation of the concentration of the gel, the temperature, and the rate of addition of the components, the grain size distribution can be controlled to meet specific requirements.

In the silver halide dispersion, gelatin molecules adsorb at the surface of the silver halide grain, surrounding the grain and forming a barrier that stabilizes the dispersion. The adsorbed layer also, in all likelihood, affects the radiation sensitivity of the grain and makes reduction by developers, more controllable. This is important in the development process and makes it possible to obtain desired results from a given system based on easily controlled parameters such as developer chemistry, development time, temperature, etc.

### Photographic Emulsion Preparation

The exact methods used in preparing commercial photographic emulsions are closely guarded trade secrets, but the basic procedures are well known. There are two general classes of emulsions, the characteristics of which are determined by the end use. They are "negative" emulsions that are used for exposure in cameras and produce a reversed or negative image, and

print images that produce the final photograph that we show off to our friends and relatives.

Negative emulsions generally must exhibit a relatively wide flexibility in terms of sensitivity since they are used under conditions that are generally beyond the control of the casual photographer. It would be rather impractical if we couldn't take our vacation pictures in bright sunlight and shade with the same camera and film. The professional photographer has the option of having several thousands of dollars of cameras dangling around her neck to suit the conditions. Most of us do not.

Print or paper materials, on the other hand, are used under highly controlled conditions of exposure and can therefore have a much more limited "range" of sensitivity and provide the user with more direct control over the final results of the development process.

The preparation of a negative emulsion involves four distinct, but interrelated, steps: (1) the formation of silver halide crystals in gelatin through a precipitation process, (2) the recrystallization of the silver halide grains by physical or Ostwald ripening, (3) a washing process that removes excess soluble salts from the emulsion, and (4) a digestion or chemical sensitizing process involving the heating of the emulsion to increase its sensitivity to incident light.

The silver halide is formed through the reaction of a halide and ammoniacal silver nitrate, in a dilute (approximately 1.5 percent) solution of gelatin at a temperature between 45° and 70° C. The halide and the silver solutions may be added to the gelatin together, in what is termed a double-jet process, or separately (single-jet), in which case the halide is added first followed by the silver nitrate solution. The concentration of gelatin, the temperature, the concentrations of the two solutions and the rates of addition are important factors in determining both the average size and the size-distribution of the dispersion of silver halide and all must be carefully controlled.

After crystallization, the emulsion is stored for several hours at a moderate temperature during which the average crystal size increases via Ostwald ripening in which the smaller crystals tend to dissolve while the larger crystals grow as crystallization nuclei.

Following this ripening process the by-products are removed and the amount of free halide is reduced. Historically this was accomplished by chilling the emulsion to a gel and forcing it through a perforated screen to form "noodles" which were then washed in running water. Other methods, which are generally trade secrets, but include membrane filtration techniques, are now used in some cases.

After washing, additional gelatin is added to bring the emulsion to its final gelatin composition. Quite often, the added gelatin is rich in sulfur containing amino acids. The final emulsion is then heated to a temperature between 50° and 80°C for about an hour to facilitate the interaction of the sulfur in the added gelatin and the silver crystals. The sulfur-silver halide interaction increases the number and size of the silver sulfide sensitivity centers and improves the characteristics of the grains. An alkaline gold thiocyanate may also be added at this stage to increase the sensitivity of the emulsion.

Other additives to the final emulsion may include:

1. stabilizers to retard changes in the size and size distribution of the grains;
2. antifogging substances to retard the development of unexposed grains of silver halide when the image is developed;
3. a gelatin hardener to prevent the gelatin from excessive swelling in processing;
4. surfactants and other components to control the wetting and other fluid characteristics of the emulsion during the coating operation;
5. surfactants, lubricants, and anti-static agents to control the surface properties of the dried emulsion;
6. color-sensitizing dyes that expand the range of light sensitivity of the emulsion.

Because the printing of images on paper is carried out in the darkroom under closely controlled conditions, the light sensitivity of the emulsion is not as critical as other visual aspects of the final print such as the tone and contrast of the image.

The emulsions used for developing papers differ from negative emulsions in a number of important respects. In preparing the paper photographic emulsion, the silver may be added to the gelatin solution containing the soluble halide, as in the preparation of a negative emulsion, the halide may be added to the gelatin silver solution, or the silver and soluble halide may be added simultaneously. The rate of addition and the concentrations involved are all designed to produce fine, uniformly sized crystals. In some processes, precipitation takes place in a slightly acid solution to inhibit recrystallization and growth of the crystal size. An excess of soluble halide is avoided for the same reason.

Paper emulsions are generally not heat ripened, as are negative systems, since that would result in larger average crystal sizes and tend to increase the sensitivity. Nor are they generally washed, because the concentration of salts is low and their presence tends to reduce further ripening in storage and changes in sensitivity. In addition, when the emulsion is coated on the paper, a

significant amount of the soluble salts is absorbed by the paper stock and thus removed from the system. Those ions that remain in the emulsion may have a desirable influence on the color or contrast of the final image.

The speed or sensitivity of an emulsion can be adjusted in many cases by the use of color-sensitizing dyes (see below). Other additives may be antiseptics, such as phenol or thymol to inhibit the growth of microorganisms; hardeners, such as alum, formaldehyde and glyoxal to improve the physical characteristics of the emulsion during storage and development; and wetting agents, such as saponin or other surfactants to reduce surface tension and facilitate the coating of the emulsion.

Special papers with wider ranges of applicability in terms of contrast, spectral sensitivity, tonal qualities, etc., may be produced in one of two ways: (1) by the admixture of two emulsions of different contrast and color sensitivity, and (2) by sensitizing in such a way that the result varies with the wavelength of the exposing light.

The silver halide grains in a paper emulsion seldom exceed 0.01 to 0.02 microns as compared with from 1.0 to 2.0 microns in a negative emulsion and the amount of silver halide in the coated paper per unit area is about one-fifth that of a negative material.

### Color Sensitizing

The radiation sensitivity of silver halides ends for all practical purposes at about 525 m $\mu$ . In Figure 3 Curve A illustrates the spectral sensitivity of a typical silver bromo-iodide emulsion and B illustrates the average human visual response curve. As the curves show, the maximum response of the eye is in the yellow-green near 550 m $\mu$  which lies beyond the sensitivity range of the emulsion, which is much more sensitive to the violet and blue than the eye.

The sensitivity of the silver halides may be extended to radiation of longer wavelengths by the addition of dyes or "color sensitizers." Although referred to as dyes, color sensitizers are not ordinary dyes in that they are not used to color cloth or other materials.



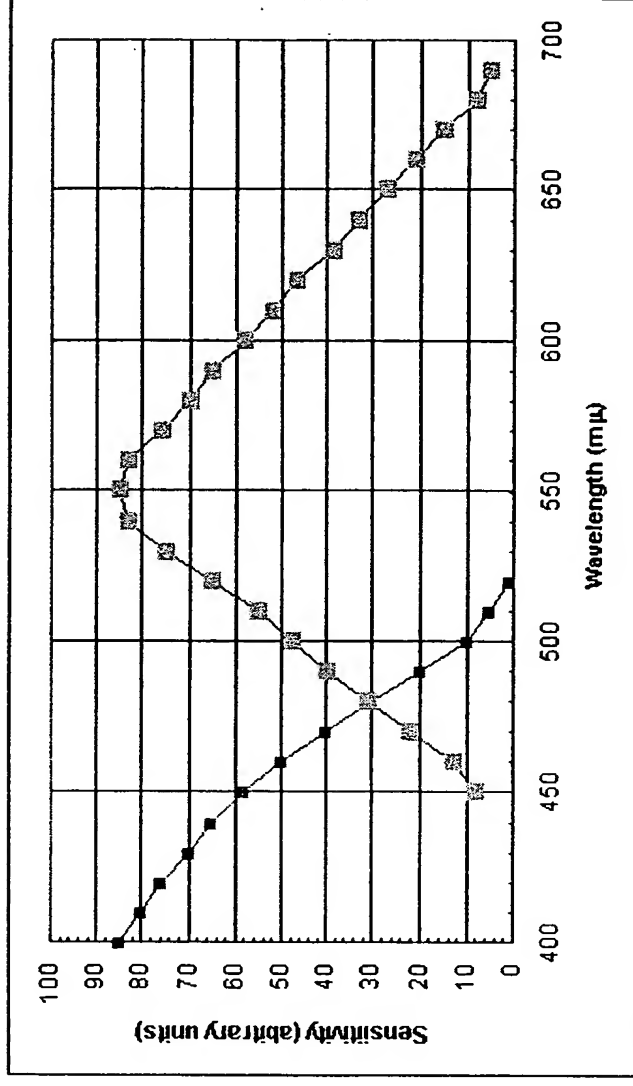


Figure 3: Curves Approximating the Light Sensitivity of Typical Silver Halide Crystals (blue) and the Human Eye (yellow)

Emulsion sensitizing results from the absorption of radiant energy by the dye at a wavelength that would not affect the silver halide, and the transfer of that "exposure" energy to the silver halide to form a latent image and make the affected grain developable. If a dye is a sensitizer, then its action depends upon the absorption characteristics of the silver halide-adsorbed color sensitizer complex, which may be quite different from the absorption of the dye itself. Since the sensitivity of such dyes varies greatly, it is often necessary to use a combination of materials to obtain a specific result. Some combinations, however, do not work well together, so that the system balance must be carefully studied before the final emulsion composition is determined. There are substances that may or may not be sensitizers themselves but greatly increase the sensitizing action of other dyes. These are known as super-sensitizers and are of considerable importance in facilitating the use of conventional sensitizing dyes. Whatever the dye used, the quantity required is always quite small.

## Development

The rate of development of the individual grains in an emulsion is affected by so many factors, such as the rate of diffusion of the solution through the gelatin matrix, the adsorption of the developing agent, the solution of the silver halide, oxidation products of the developing agent and the accumulation of restraining by-products, that exact analysis of it is difficult. The time of appearance of a visible image is, within limits, a reliable indication of the rate of development. It varies with different emulsions and is quite different with different developing agents, but the variation with temperature, dilution and pH is almost directly related to the variation in the rate of development.

The rate of development, as determined from the change in the optical density of the developed image, is complicated by the fact that density increases in two different ways: (1) by the increase in the amount of silver as the grains develop and (2) by an increase in the number of grains in the process of development. Density grows rapidly at first and then slows down until development is complete and no further growth in density takes place. Prolonged development would, of course, increase overall density through the development of unexposed grains (fog).

### Halting Development – the Stop Bath

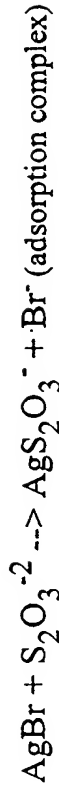
Once the exposed image has been developed to the desired degree, it is necessary to halt the chemical process quickly to prevent over development and the production of fog. The solution used to that end is referred to as the "stop" bath. Since developers function a relatively high pH's, the typical stop bath is simply a solution of acetic or some other weak acid. The action of the acid is so rapid it usually requires only seconds for the process to be effectively halted.

### The Fixing Process

Once the developed image is obtained, a large amount of unexposed and undeveloped silver halide remains in the emulsion. If that silver halide is not removed before the image is exposed to radiation capable of producing a latent image, the image will continue to darken. The process of removing the residual silver halide from the image is called "fixing."

The silver halides are only slightly soluble in water; therefore, to remove the material remaining after development it is necessary to convert it to soluble complexes which can be removed by washing. Sodium thiosulfate, commonly termed "hypo," has been used for this purpose since 1839.

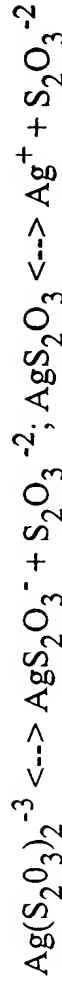
The reactions in fixing can be written as follows:



which is followed by



and by



Within limits, the rate of fixation is indicated by the clearing time, i.e., the time required to remove all visible traces of silver halide from the image. This time depends on the concentration of thiosulfate, the temperature, the agitation of the solution, but more particularly on the emulsion and the extent to which the fixing bath has been used. Fine-grain emulsions fix in less time than those of larger grains, and paper emulsions of silver chloride fix faster than bromo-iodide negative emulsions. Thickly coated films, other things being equal, fix more slowly than those with a thin emulsion coating. The fixing time increases appreciably as the solution becomes depleted. With continued use the halide-ion concentration rises in proportion to the amount of silver halide dissolved. When the product of the silver-ion and the halide-ion activities reaches the solubility product of the least soluble silver halide present, the solution will dissolve no more of that silver halide and fixation will necessarily be incomplete.

It is usually desirable to harden the gelatin after development, and while this may be accomplished by a hardening stop bath prior to fixing, the usual practice is to combine hardening with fixing. The conventional fixing and hardening bath contains in addition to the fixing agent:

1. An organic acid, usually acetic, to provide the necessary acidity to stop development and create the proper pH for effective hardening.
2. Sodium sulfite, which prevents the decomposition of the thiosulfate by the acid and forms colorless oxidation products of the developer thus preventing staining.

### 3. Alum as a hardening agent.

The hardening produced by alum is due to the reaction of the aluminum ions,  $Al^{+3}$ , and the carboxyl groups of the gelatin with the formation of cross-linkages between chain molecules. The degree of hardening, other things such as temperature, alkalinity of the film when placed in the fixing bath, etc., being equal, depends on the pH of the solution which in turn depends on the relative proportions of acid, sulfite and alum.

Since the addition of developer tends to increase the pH of the fixing bath, the solution should be buffered against an increase in pH. For this reason weak organic acids, such as acetic acid, are used in preference to a stronger acid, such as sulfuric. The addition of boric acid increases the useful hardening life of potassium alum baths and reduces the tendency of the bath to form a sludge.

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